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# Waste bleaching clays as fillers in hot bituminous mixtures



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#### HIGHLIGHTS

- Use of waste bleaching clays as fillers in HMA for binder courses.
- Substitution of limestone filler with waste bleaching clays from food industry.
- The use of bentonite fillers does not worsen the compatibility of mixes.
- Digested spent bentonite clay determines an increase of HMA stiffness values.
- Digested spent bentonite clay raises the permanent deformations resistance of HMA.

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#### ABSTRACT

Today, it is a well known fact that mineral fillers have a key role in controlling the mechanical characteristics of hot mix asphalts. The cohesion properties of bituminous mastics depend upon the fillers used, and, therefore, so do the effective strength of asphalt concrete and the durability of the pavement layer.

In this study, waste bleaching clays from the food industry are proposed as an alternative to common limestone mineral filler for the production of Hot Mix Asphalts (HMA). The bleaching clays used here come from two consecutive stages in the industrial process for decolouring vegetable oils and producing biogas from waste clay, where the former is richer in residual organic fats (20–25% in weight against less than 1%).

The aim of this research was to assess the performance of a common binder course asphalt mixture, in terms of physical and mechanical characteristics, when waste bleaching clays are used as an alternative. For this purpose, data obtained from a control hot mix asphalt produced with traditional limestone filler were compared to those recorded for bituminous samples containing waste bleaching clay from either the first or the second stage above, in place of the limestone filler.

Results show a significant difference between the effects produced by the bleaching clays from the two stages on the performance of the asphalt concrete, where the second stage shows more positive effects both on indirect tensile strength and stiffness and on the bituminous mixture's resistance to permanent deformations.

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## 1. Introduction

Traditionally, Hot Mix Asphalts (HMA) contain two components: aggregates and bituminous binders. While the former basically act as a lithic skeleton for the material, bitumen acts as a binder for all the elements in the mixture. In more detail, the fraction of aggregates sieved through a 63  $\mu m$  sieve (EN 13043), together with the mineral filler, which is generally added to the

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lithic mixture during the process, are combined with the binder to form a bituminous mastic. This resulting mastic is capable of affecting the physical and mechanical properties of the mixture to a considerable degree [1]. For this reason, the study of the materials that make up the bituminous mastic, and the way they influence its performance, are of pressing interest to asphalt producers and users.

The ASTM D-242 standard states that mineral fillers should consist of "finely divided material matter" (for example rock dust) sufficiently dry and free from agglomerations [2]. In practical applications, fillers are generally considered to be material with a granulometry of less than 75  $\mu$ m [3]. Over the years, various materials have been both studied and used as fillers in HMA. Fill materials can be "natural" (i.e. rock dust) or "imported" (i.e. Portland

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cement, lime, ash and waste) [4]. Currently, topics such as saving energy and resources, and the ever-growing interest of civil society in issues relating to environmental protection, have concentrated attention on the potential re-use of various kinds of industrial waste in road construction [5]. There are now many studies on scrap and/or waste materials used in addition or in substitution to raw materials in the production of asphalt concretes [6]. The work presented in this article can be set within this context, insomuch as it covers the use of spent bentonite bleaching clays, due to go to landfill, as substitutes for the traditional limestone filler used in the preparation of hot asphalt mixes. Its re-use has a twofold environmental benefit, since it saves natural resources such as limestone fillers, and reduces the volume of materials disposed of in landfills.

Bleaching clays are aluminium hydrosilicates containing small amounts of Mg, Ca and Fe, which are similar to normal clays, but more hydrated, and have the peculiarity of being able to remove coloured impurities from mineral, vegetable and animal oils, and from other liquids [7]. They are divided into Floridinic clays, which are active in the natural state, and bentonite clays, active only after chemical treatment. The main component of bentonite is montmorillonite, which is a clayey mineral belonging to the class of phyllosilicates called smectites. The special properties found in bentonite clays, especially their adsorption and binding capacities, make them particularly suitable for use in sectors such as the ceramic industry, the food industry, the foundry and smelting sector, water treatment [8] and, indeed, many others.

In the case under examination, the bentonite clays are waste from the food industry, left over from the process of bleaching vegetable oils. Two different types of bentonite clays were used, in the form of waste from two consecutive industrial processes: spent bentonite (labelled Ut) obtained from a vegetable oil bleaching process, and digested spent bentonite (labelled Ud) the result of the anaerobic digestion of spent bentonite within a reactor producing biogas. While many works have involved using spent bentonite clay in various sectors, there is no consolidated scientific literature covering of this industrial waste used as a filler for HMA concrete.

## 2. Materials and test methods

## 2.1. Aggregates and bitumen

In this study, three different binder layer HMA mixtures were prepared. For two of them, Ut or Ud fillers totally substituted the weight of the traditional limestone filler, which was used in the reference mixture. The granulometric curve and the filler and binder percentages required were defined during the mix design phase (Fig. 1). The content of bitumen was fixed at 5.8% in weight for the aggregate mixture, while the percentage of filler to be used, whether limestone or from spent bleaching clays, was fixed at 3% in weight for the aggregates. The choice of grain size was based on the typical gradation limits of HMA mixtures for binder layers. Common limestone crushed stones were used as the aggregate, together with

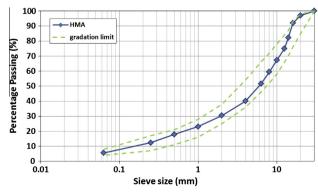


Fig. 1. Aggregates gradation.

around 22% Reclaimed Asphalt Pavement (RAP). The properties of the aggregates are given in Table 1. The volumetric mass of the aggregates was calculated according to the EN 1097-6 standard.

A common 50/70 penetration grade bitumen was used as a binder; its rheological properties are given in Table 2.

#### 2.2. Filler from decolouring clay

Two different bentonite fillers were used in the experimental study (Fig. 2), obtained from two consecutive phases in the process of bleaching vegetable oils and producing energy from biogas. The filler labelled Ut was derived directly from the bleaching phase and, for this reason, its oil content is between 20% and 25% of its dry weight. The other filler, Ud, i.e. the digested spent bentonite, is the result of the anaerobic digestion of the Ut filler during the process to produce biogas. This phase of biochemical conversion determines a reduction of the content of residual oils, to below 1%.

In order to be used as fillers, both spent bleaching clays were characterized geometrically through a granulometric analysis (EN 933-10) and volumetrically, determining volumetric mass (EN 1097-7) and Rigden Voids (EN 1097-4). Using this test, it was possible to determine the inter-granular porosity of the dry, compacted filler.

From the granulometric analysis, it emerged that 100% of the material of both fillers can pass through a 63  $\mu$ m sieve, which means that both the spent bleaching clays, from a granulometric perspective, can be placed in the category of fillers for road use. The results of the volumetric analysis and the determination of Rigden Voids are given in Table 3, where the values are compared to those obtained with a limestone filler.

From the comparison of their volumetric characteristics, a clear difference emerges, in terms of density, between the spent bentonite clays and the limestone filler. When comparing the Rigden Voids (RV), the value of the Ud filler is 22% above that of the filler of reference, whose RV value is strictly comparable to that of the Ut filler. This element is extremely important because, according to Cooley, the percentage of Rigden Voids is fundamental in defining the stiffness of the bitumen mastic [9] as, the higher the voids, the greater the quantity of fixed bitumen.

#### 2.3. Test methods and experimental program

Three different mixtures were prepared to evaluate the volumetric and mechanical characteristics of the mixtures where the bentonite filler had been totally substituted to the traditional limestone filler. Of these, the first contained Ut filler (HMAUt), the second Ud filler (HMAUd) and the third was a traditional binder used for comparison (HMAF). Seven specimens were prepared for each mixture, each with a weight of 4500 g and a diameter of 150 mm, using a gyratory compactor and in accordance with the ASTM D6925 standard, with a compaction pressure of 600 kPa, external slope angle of 1.25° and 180 revolutions. The mixtures were compacted at a processing temperature of 180 °C. All the tests were carried out on fully cured specimens.

The experiment was divided into three phases.

The working and volumetric characteristics of the mixtures were defined in the initial prequalifying phase, by analysing the compaction curves obtained from the gyratory compactor. This analysis step was validated by an Indirect Tensile Strength (ITS) test at 25 °C. In line with the EN 12693-23 standard, a constant velocity of 50 mm/min was applied to the specimen until it broke. The objective of the analysis step was to evaluate the influence, in terms of compaction, of the spent bentonite clay contained within the bitumen mixture where the limestone filler has been totally substituted.

 Table 1

 Designation and composition of HMA concrete with limestone filler.

Material	Density (g/cm <sup>3</sup> )	Mixture (%)
Crushed stones 6–22	2.67	49
RAP 0-10	2.63	22
Sand 0–6	2.68	26
Limestone filler	2.66	3

**Table 2** Properties of asphalt 50/70 pen binder.

	Unit	Characteristic value	Standard
Penetration @ 25 °C	dmm	50-70	EN 1426
Soft. Point	°C	50	EN 1427
Dynamic Visc. @ 60 °C	Pa s	≥145	EN 12596
Fraass	°C	-8	EN 12593

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